

A3 recovering from the organic liquid a product comprising a mixture of organic compounds containing less sulfur and less nitrogen than the hydrotreated petroleum distillate.

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REMARKS

This Amendment is respectfully submitted to place rejected claims of subject Application in condition for allowance. In particular, Claims 8, 15 and 19 to 21 inclusive have been amended to more particularly point out and distinctly claim the subject matter of the invention.

As urged by Examiner, the term "the high-boiling oxidation feedstock" has been replaced with the term "the hydrotreated petroleum distillate" in the last line of Claim 21.

Claims 8 and 19 have been amended to further recite the steps of treating the immiscible aqueous phase separated from the reaction mixture to remove at least a portion of the sulfur-containing and/or nitrogen-containing organic compounds, recycling the treated aqueous phase to the reaction mixture. Support for these amendments is found in the specification of subject application, for example at page 27, lines 17 to 32, and Claims 6 and 14.

Claim 19 has been amended to recite components containing less than about 50 parts per million of sulfur. Support for this amendment is found in the specification of subject application, for example at page 31, lines 25 to 28.

Claim 15 has been amended to recite a solvent having a dielectric constant in a range from about 24 to about 80. Support for this amendment is found in the specification of subject application, for example at page 13, lines 17 to 18.

The term "hydroxide" has been deleted from Claim 20. Support for this amendment is found in the specification of subject application, for example at page 13, lines 25 to 26.

5 Claim 21 has been amended to recite sulfur-containing and ~~nitrogen-containing~~ organic compounds. Support for this amendment is found in the specification of subject application, for example at page 14, lines 11 to 14.

### 112 Rejections

10 In outstanding Office Action, Claim 21 was rejected under 35 U.S.C. § 112 as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. Applicants respectfully traverse this rejection.

15 In view of the amendments and remarks submitted above, it is the position of Applicants that instant Claim 21 meets all requirements of 35 U.S.C. § 112.

### 103 Rejections

20 In outstanding Office Action, Claims 1 to 15, 17 to 19, and 21 were rejected under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No 6,217,748 in the name of Shigeto Hatanaka, Osamu Sadakane, and Hideshi Iki (Hatanaka et al.), in view of U.S. Patent No 6,402,939 in the name of Teh Fu Yen, Hai Mei, and Steve Hung-Mou Lu, (Yen et al.) and U.S. Patent No 3,341,448 in the name of  
25 John Frederick Ford, Timothy Arrowsmith Rayne and Dennis George Adlington (Ford et al.). Applicants respectfully traverse this rejection.

The Hatanaka et al. reference of record describes a four-step hydrodesulfurization (HDS) of a sulfur-containing petroleum hydrocarbon diesel gas oil. The gas oil is catalytically hydrodesulfurized at 300° C. to about 500 ppm sulfur in a first HDS  
5 step. Step two is distillation at a cut point temperature in a range of 320 to 340° C. Step three is a second HDS at 320 to 380° C., but of only the heavy fraction of the distillation. The twice hydrodesulfurized heavy fraction (90 ppm sulfur) is mixed with the light fraction of the distillation (10 ppm sulfur) to a sulfur  
10 content of up to 0.01% (50 ppm sulfur, Hatanaka et al. Example 3).

Obvious disadvantages of the Hatanaka et al. process include the costs of the additional hydrogen and energy required for both the distillation and further hydrodesulfurization steps. While Hatanaka et al. state the hydrogen sulfides from the two HDS steps  
15 may be removed from the off-gas by an amine absorption arrangement, carbon oxides are also by-products of the HDS steps. Generation of the additional hydrogen also produces more carbon dioxide. These are especially critical at a time when most industries are striving to limit the amount of emitted "green house"  
20 gas such as carbon dioxide.

By contrast Applicants' novel process includes a catalyzed, hydrogen peroxide, promoted oxidative desulfurization of a sulfur-rich, mono-aromatic-lean fraction without further production of carbon dioxide or hydrogen sulfides. An additional advantage of  
25 Applicants' novel oxidative desulfurization process is that oxidation is selective to sulfur and nitrogen containing heteroaromatic compounds, i.e. does not saturate the non-sulfur, non-nitrogen multi-ring aromatic hydrocarbons.

The Yen et al. reference of record describes oxidizing sulfur  
30 compounds in fuels to sulfones with a hydroperoxide in an aqueous-organic medium while it subjected ultrasound.

A declaration under 37 CFR § 1.131 of prior invention by Applicants, identified as Paper No. 7, is hereby presented to further the prosecution of subject Application. In particular, the declaration is a showing of facts which establish that reduction to  
5 practice of Applicants' invention, as described and claimed in subject application, was completed in the United States of America and the United Kingdom prior to September 28, 2000, the filing date of the application from which U.S. Patent No. 6,402,939 (Yen et al.) matured.

10 Applicants respectfully request Primary Examiner Griffin to withdraw rejections under 35 U.S.C. § 103 (a) which rely upon the Hatanaka et al. reference in combination with the Yen et al.

The Ford et al. reference of record, which claimed priority, application Great Britain, November 24, 1961, is directed to a  
15 desulphurization of a petroleum residue containing a least a portion of material boiling above 250° C. and having at least 1 percent by weight of sulfur as sulfur-containing compounds by two successive stages. Their first stage is a combination of an oxidative step and a thermal desulphurization step, and the other stage is a  
20 hydrocatalytic desulphurization of the total effluent from the first stage.

After contacting the feedstock with molecular oxygen or air as the oxidizing agent, preferably with solid metal promoter at 130 to 180° C. for 2 to 20 hours, any oxidized sulfur compounds are  
25 said to be eliminated as gaseous decomposition products from the reaction mixture by thermal decomposition at temperatures of up to 400° C. within 1/2 to 5 hours. Clearly the gaseous decomposition products of the Ford et al. process are a potential source of atmospheric pollution.

30 At best Ford et al. report their first stage removed only 30 percent of the sulfur based on untreated feedstock. While their hydrocatalytic desulphurization stage further removed 30 percent,

their process removed only 60 percent of the sulfur based on untreated feedstock. Feedstock of at least 1 percent by weight of sulfur treated by the first of Ford et al. is expected to contain at least 0.7 percent of sulfur (7,000 ppm), and the overall two stage  
5 process of Ford et al. is expected to contain at least 0.4 percent of sulfur (4,000 ppm).

While in the past a typical specifications for Diesel fuel was a maximum of 0.5 percent by weight, by 1993 legislation in Europe and United States limited sulfur in Diesel fuel to 0.3 weight  
10 percent. By 1996 in Europe and United States, and 1997 in Japan, maximum sulfur in Diesel fuel was reduced to no more than 0.05 weight percent. This world-wide trend must be expected to continue to even lower levels for sulfur.

Rather than disclosing or suggesting the novel process of  
15 Applicants, the Ford et al. reference of record teaches a process which became obsolete after 1993.

Available evidence strongly suggests that ultra-low sulfur fuel is a significant technology enabler for catalytic treatment of diesel exhaust to control emissions. Fuel sulfur levels of below 15  
20 ppm, likely, are required to achieve particulate levels below 0.01 g/bhp-hr. Such levels would be very compatible with catalyst combinations for exhaust treatment now emerging, which have shown capability to achieve NOx emissions around 0.5 g/bhp-hr. Furthermore, NOx trap systems are extremely sensitive to fuel  
25 sulfur and available evidence suggests that they need would need sulfur levels below 10 ppm to remain active. Therefore, a critical element of Applicants' novel process is reduction of nitrogen.

By contrast, working Examples of Applicants' novel process demonstrated that their process removed more than 95 percent of  
30 the of the sulfur and nitrogen (See Example 3 where 99.3 percent sulfur was removed, i.e., from 48 ppm sulfur and 49 ppm nitrogen to 0.3 ppm sulfur and 2.5 ppm nitrogen, Example 4 where 97.5

percent sulfur was removed, i.e., from 24 ppm sulfur and 16 ppm nitrogen to 0.3 ppm sulfur and 2.5 ppm nitrogen, and Example 5 where 99.4 percent sulfur was removed, i.e., from 70 ppm sulfur to 0.4 ppm sulfur).

- 5 Applicants respectfully request Primary Examiner Griffin to withdraw all rejections under 35 U.S.C. § 103 (a) which rely upon the Hatanaka et al. reference in combination with the Ford et al.

10 Claims 16 and 20 were rejected under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No 6,217,7480 (Hatanaka et al.), in view of U.S. Patent No 6,402,939 (Yen et al.) and U.S. Patent No 3,341,448 (Ford et al.) as applied to Claims 15 and 17 above, and further in view of U.S. Patent No 2,749,284 in the name of Dennis Alexander John Noble (Noble). Applicants respectfully traverse this rejection.

15 The Noble reference of record, which claimed priority, application Great Britain, July 4, 1951, is describes a process by which 25.9 percent of the sulfur was removed from a gas oil containing 1.16 percent total sulfur by oxidation with small amounts of an aqueous phase and extraction with methanol. The  
20 product contained 0.84 percent of sulfur (860 ppm sulfur). Contrary to the position of Examiner, these results of Noble do not suggest the effective removal of oxidized sulfur (AND nitrogen) compounds demonstrated in Applicants' novel process.

25 Applicants respectfully request Primary Examiner Griffin to withdraw all rejections under 35 U.S.C. § 103 (a) which rely upon the Hatanaka et al. reference in combination with the Noble and Ford et al. references of record.

30 Rather than disclosing or suggesting the novel process of Applicants, the Noble reference of record teaches a process which became obsolete after 1993. At most, the Noble and Ford et al. references of record in combination establish a present need for an

improved process for desulphurization of blending components for diesel fuel.

Applicants agree with Examiner that the references of record but not applied do not disclose or suggest Applicant's novel subject matter which includes, for example,

- (i) hydrotreating a petroleum distillate consisting essentially of material boiling between about 50° C. and about 425° C. by a process which includes reacting the petroleum distillate with a source of hydrogen at hydrogenation conditions in the presence of a hydrogenation catalyst to assist by hydrogenation removal of sulfur and/or nitrogen from the hydrotreated petroleum distillate;
- (ii) fractionating the hydrotreated petroleum distillate by distillation to provide at least one low-boiling blending component consisting of a sulfur-lean, mono-aromatic-rich fraction, and a high-boiling oxidation feedstock consisting of a sulfur-rich, mono-aromatic-lean fraction;
- (iii) contacting the high-boiling oxidation feedstock with a soluble quaternary ammonium salt containing halogen, sulfate, or bisulfate anion, and an immiscible aqueous phase comprising a source of hydrogen peroxide, and at least one member of the group consisting of phosphomolybdic acid and phosphotungstic acid, in a liquid reaction mixture under conditions suitable for reaction of one or more of the sulfur-containing and/or nitrogen-containing organic compounds;
- (iv) separating from the reaction mixture an essentially organic liquid and at least a portion of the immiscible aqueous phase; and
- (v) treating at least a portion of the recovered organic liquid with a solid sorbent, an ion exchange resin, and/or a suitable immiscible liquid containing a solvent or a

soluble basic chemical compound, to obtain a product containing less sulfur and/or less nitrogen than the oxidation feedstock.

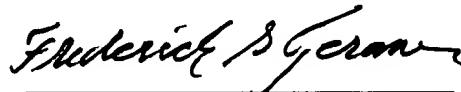
5 Applicants urge that Claims 1 to 21, all claims now presented, are in condition for allowance. Applicant respectfully requests Examiner to pass subject application for allowance.

10 Do not hesitate to contact Frederick S. Jerome whose telephone number is (630) 832-7974 (FAX (630) 832-7976) if additional assistance is needed regarding this paper or earlier papers for Applicants.

Applicants and their undersigned Attorney appreciate Examiner's attention and further consideration of this matter.

Respectfully submitted,

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Version of Amended Claims with Markings  
to Show Changes Made

8. (Once amended) The process according to claim 7  
5 further comprising treating the immiscible aqueous phase  
separated from the reaction mixture to remove at least a portion of  
the sulfur-containing and/or nitrogen-containing organic  
compounds, recycling the treated aqueous phase to the reaction  
mixture, and blending at least a portion of the low-boiling blending  
10 component with the product containing less sulfur and/or less  
nitrogen than the high-boiling oxidation feedstock to obtain a  
component for refinery blending of transportation fuel.

15. (Once amended) The process according to claim 12  
wherein the treating of recovered organic liquid includes use of at  
15 least one immiscible liquid comprising a solvent having a dielectric  
constant in a range from about 24 to about 80 suitable to  
selectively extract oxidized sulfur-containing and/or nitrogen-  
containing organic compounds.

19. (Once amended) The process according to claim 7  
20 further comprising treating the immiscible aqueous phase  
separated from the reaction mixture to remove at least a portion of  
the sulfur-containing and/or nitrogen-containing organic  
compounds, recycling the treated aqueous phase to the reaction  
mixture, and blending at least a portion of the low-boiling fraction  
25 with the product containing less sulfur and [or] less nitrogen than  
the oxidation feedstock to obtain components containing less than  
about 50 parts per million of sulfur for refinery blending of a  
transportation fuel.

20. (Once amended) The process according to claim 17  
30 wherein the treating of recovered organic liquid includes use of at  
least one immiscible liquid comprising an aqueous solution of a

soluble basic chemical compound selected from the group consisting of sodium, potassium, barium, calcium and magnesium in the form of [hydroxide,] carbonate or bicarbonate.

21. (Once amended) A process for the production of refinery transportation fuel or blending components for refinery transportation fuel, which process comprises:

hydrotreating a petroleum distillate consisting essentially of material boiling between about 50° C. and about 425° C. by a process which includes reacting the petroleum distillate with a source of hydrogen at hydrogenation conditions in the presence of a hydrogenation catalyst to assist by hydrogenation removal of sulfur and [or] nitrogen from the hydrotreated petroleum distillate;

contacting the hydrotreated petroleum distillate with a soluble quaternary ammonium salt containing halogen, sulfate, or bisulfate anion, and an immiscible aqueous phase comprising a source of hydrogen peroxide, and at least one phospho-metallic acid, in a liquid reaction mixture under conditions suitable for reaction of one or more of the sulfur-containing and nitrogen-containing organic compounds;

separating from the reaction mixture both an essentially organic liquid and at least a portion of the immiscible aqueous phase; and

recovering from the organic liquid a product comprising a mixture of organic compounds containing less sulfur and [or] less nitrogen than the hydrotreated petroleum distillate [high-boiling oxidation feedstock].